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## **Fabrication and Characterization of Samples for a Material Migration Experiment on the Experimental Advanced Superconducting Tokamak (EAST)**

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# **Fabrication and Characterization of Samples for a Material Migration Experiment on the Experimental Advanced Superconducting Tokamak (EAST)**

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## **Abstract**

This report documents work done for the ITER International Fusion Energy Organization (Sponsor) under a Funds-In Agreement FI 011140916 with Sandia National Laboratories. The work consists of preparing and analyzing samples for an experiment to measure material erosion and deposition in the EAST Tokamak. Sample preparation consisted of depositing thin films of carbon and aluminum onto molybdenum tiles. Analysis consists of measuring the thickness of films before and after exposure to helium plasma in EAST. From these measurements the net erosion and deposition of material will be quantified. Film thickness measurements are made at the Sandia Ion Beam Laboratory using Rutherford backscattering spectrometry and nuclear reaction analysis, as described in this report. This report describes the film deposition and pre-exposure analysis. Results from analysis after plasma exposure will be given in a subsequent report.

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# **1. STATEMENT OF WORK**

This report documents work done for the ITER International Fusion Energy Organization (Sponsor) under a Funds-In Agreement FI 011140916 with Sandia National Laboratories.

The purpose of this work is to obtain data for validation of codes which are used to predict the extent of erosion and deposition of material that will occur in ITER. This information could impact plans for operation of ITER, for example procedures for control of tritium retention and frequency of replacement of plasma-facing components. The experiment is a collaboration between the ITER organization (IO) in France, ASIPP in Hefei China and Sandia. The principle investigators are Greg deTemmerman at the IO, Rui Ding at ASIPP and W.R. Wampler at Sandia. The primary roles of the three organizations in this collaboration are as follows. ASIPP provides samples consisting of a set of 12 machined molybdenum tiles, plasma exposure of the samples in the Experimental Advanced Superconducting Tokamak (EAST) using the Material and Plasma Evaluation System (MAPES), and computational simulations of erosion and deposition. Sandia provides preparation and analysis of samples to quantify erosion and deposition. The IO provides overall coordination of the experiment. The US, France and China are all partners in the ITER project [1].

The statement of work is as follows.

Sandia Corporation, for and in consideration of the funding provided by the Sponsor, shall perform the following work for the Sponsor:

## **I PROJECT SCOPE:**

The present technical specifications call for the preparation and characterization of a dedicated MAPES probe head consisting of molybdenum tiles to be provided by ASIPP. Aluminum and carbon layers will be deposited onto these tiles at Sandia and erosion and deposition of material due to exposure in the EAST tokamak will be determined by ion beam analysis at the Sandia Ion Beam Laboratory before and after exposure in EAST.

## **II TECHNICAL CONTENT:**

The method to be used will be similar to that used in a previous experiment described in journal publications [2,3]

## **III DELIVERABLES:**

1. Prepare coatings of carbon and aluminum on molybdenum tiles and characterize the coating thickness by ion beam analysis. Report results of film characterization to the sponsor (ITER Organization). Send the coated tiles to ASIPP for exposure in EAST.
2. After exposure in EAST, determine the erosion and deposition of material by ion beam analysis and report the results to the sponsor.

This report documents completion of the first deliverable.

## **2. FILM DEPOSITION AND INITIAL CHARACTERIZATION**

Samples for the EAST/MAPES material migration experiment, consisting of a set of 12 machined Molybdenum tiles, were received from ASIPP. Carbon and Aluminum films were deposited on the tiles as requested by the sponsor. The regions of deposition are illustrated in figure 1. The C and Al coatings were applied by magnetron sputter deposition at the Sandia Thin Film Deposition Laboratory. The method of coating was similar to that used in a previous experiment [2,3]. A layer of titanium about 50 nm thick was deposited on the Mo substrate before deposition of the C and Al films to promote adhesion. The thickness of the films was measured at 60 locations by Rutherford backscattering spectrometry (RBS) using 1.5 MeV protons at the Sandia Ion Beam Laboratory. The tiles are now ready for return to ASIPP where they will be exposed to fusion plasma in the EAST tokamak. After exposure to the plasma, the tiles will be sent back to Sandia where the film thickness will again be measured. From the change in film thickness, the amount of erosion and redeposition of material by the plasma will be determined. This information will be used to validate models being used to predict erosion and deposition of material in ITER.

The film thickness was measured by RBS using 1.5 MeV protons. Figures 2 and 3 show examples of RBS spectra. The red dashed curve shows a spectrum from Mo with no film. The

edge near channel 345 is from protons scattering at the Mo surface. When films are present protons lose energy in the film, causing a shift  $\Delta E$  of the Mo edge to lower energy. The film thickness is determined from the magnitude of this shift, which is approximately proportional to the film thickness. The black dashed curves show spectra from simulations using the SIMNRA code [4]. RBS gives the film thickness in units of number of atoms, or mass, per unit area, which can be converted to physical thickness by dividing by the density of the material. The mean value and standard deviation of the film thickness for each tile is listed in table 1. The C and Al films are both about 2.2 microns thick. The precision of the thickness measurement is about 20nm or about 1% of the film thickness.

During plasma exposure, the center four tiles (CDIJ) will be recessed and hence will be shadowed from direct contact with the plasma. These tiles were left uncoated to enable more sensitive measurements of deposition of material from the plasma exposure. RBS can provide a measurement of the thickness of deposited material, from the shift in the Mo edge due to the energy loss in the film, as illustrated in figure 4. However, the deposited material may include both C and Al. Individual coverage of C and Al can be determined by RBS from their scattering yields or peak area. However, the large background of counts from the Mo substrate, limits the sensitivity of this method to about  $10^{17}$  atoms/cm<sup>2</sup> or 100 nm. The energy loss method has a detection limit about an order of magnitude lower, but does not give the composition. We propose to use nuclear reaction analysis (NRA) with the  $^3\text{He}(^{12}\text{C},p)^{14}\text{N}$  nuclear reaction, to measure carbon coverage when the deposits are too thin for measurement by RBS. With NRA there is no background from the Mo substrate. NRA with He3 has been extensively used for quantitative analysis of light elements at the Sandia IBL. Figure 5 shows NRA spectra taken on tile C and on a thin-film carbon reference sample, using an analysis beam of 2.5 MeV  $^3\text{He}$ . The carbon coverage is determined from the proton yield (i.e. peak area) relative to the yield from a thin-film reference sample with known carbon coverage. This measurement showed that the uncoated tile has  $10^{17}$  atoms/cm<sup>2</sup> of carbon, corresponding to about 10 nm. This is in the “as received” condition. This result also shows that the detection limit for C coverage by this method is about  $10^{15}$  atoms/cm<sup>2</sup>, which is about one monolayer. Based on this result the uncoated tiles (C D I J) will be cleaned and re-measured to reduce the baseline carbon coverage for measurement of deposition by the EAST plasma. Another benefit to using  $^3\text{He}$  NRA is that the deposition of other light elements, including deuterium, Li, Be, B, O, can be determined from

the same spectra from the proton yields for similar nuclear reactions, which give peaks at other energies in the spectrum. After cleaning the uncoated tiles, the twelve tiles will be shipped to ASIPP for exposure to helium plasma in EAST, after which they will be returned to the Sandia IBL for measurement of erosion/deposition. This will be done by measuring the thickness of films and deposited material using the same methods as used for the pre-exposure analysis described above. Net erosion and deposition will be determined from the post- exposure thickness minus the pre-exposure values. It is expected that the final results of the experiment will be published in the open scientific literature.



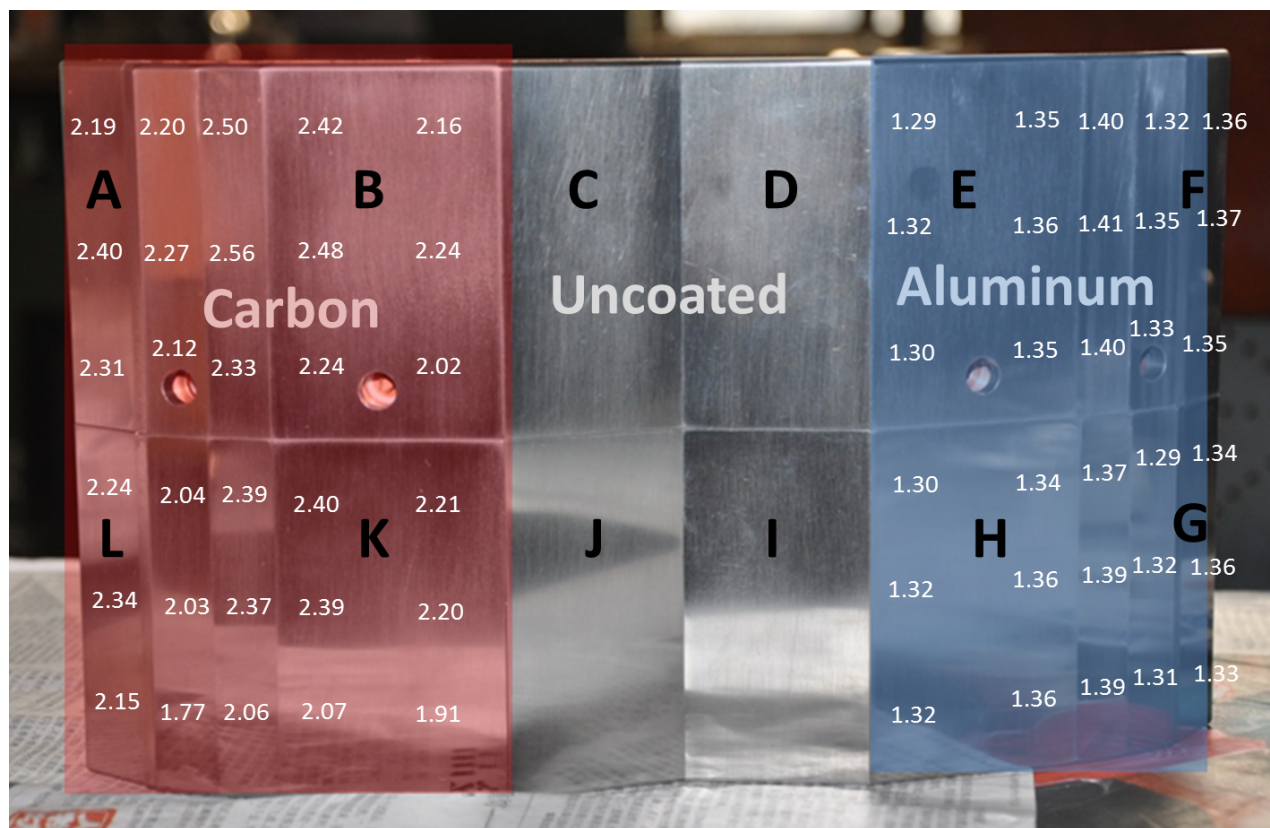


Figure 1. Mo tiles for the EAST/MAPES material migration experiment. Four tiles (ABKL) were coated with carbon and four tiles (EFGH) were coated with aluminum. The central four tiles (CDIJ) were not coated. The numbers indicate the film thickness (in units of  $10^{19}$  atoms/cm<sup>2</sup>) at 60 locations where it was measured. The overall dimensions of the assembly are 18x27 cm. Red and blue colors are to illustrate regions with carbon and aluminum coatings.

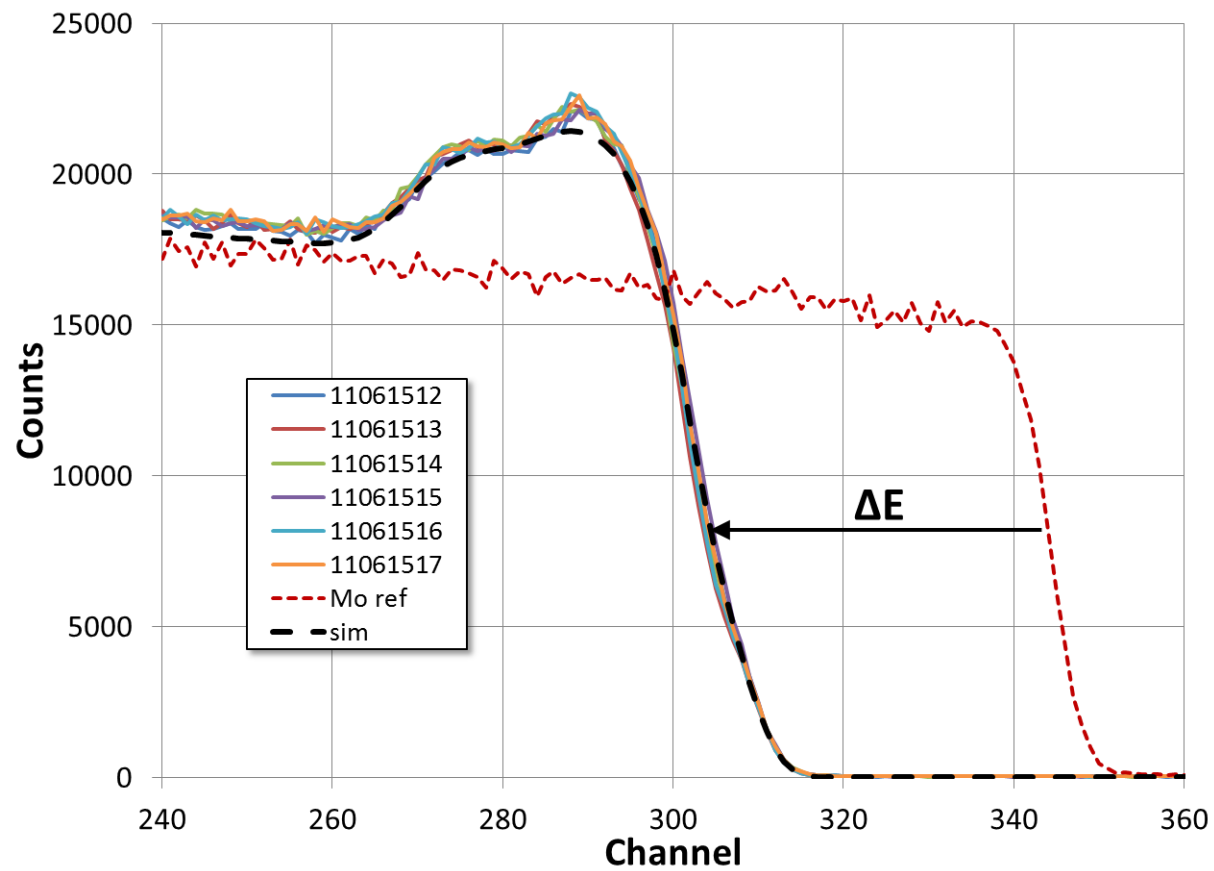


Figure 2. RBS spectra for aluminum coated tiles F & G. Channel number is proportional to particle energy.

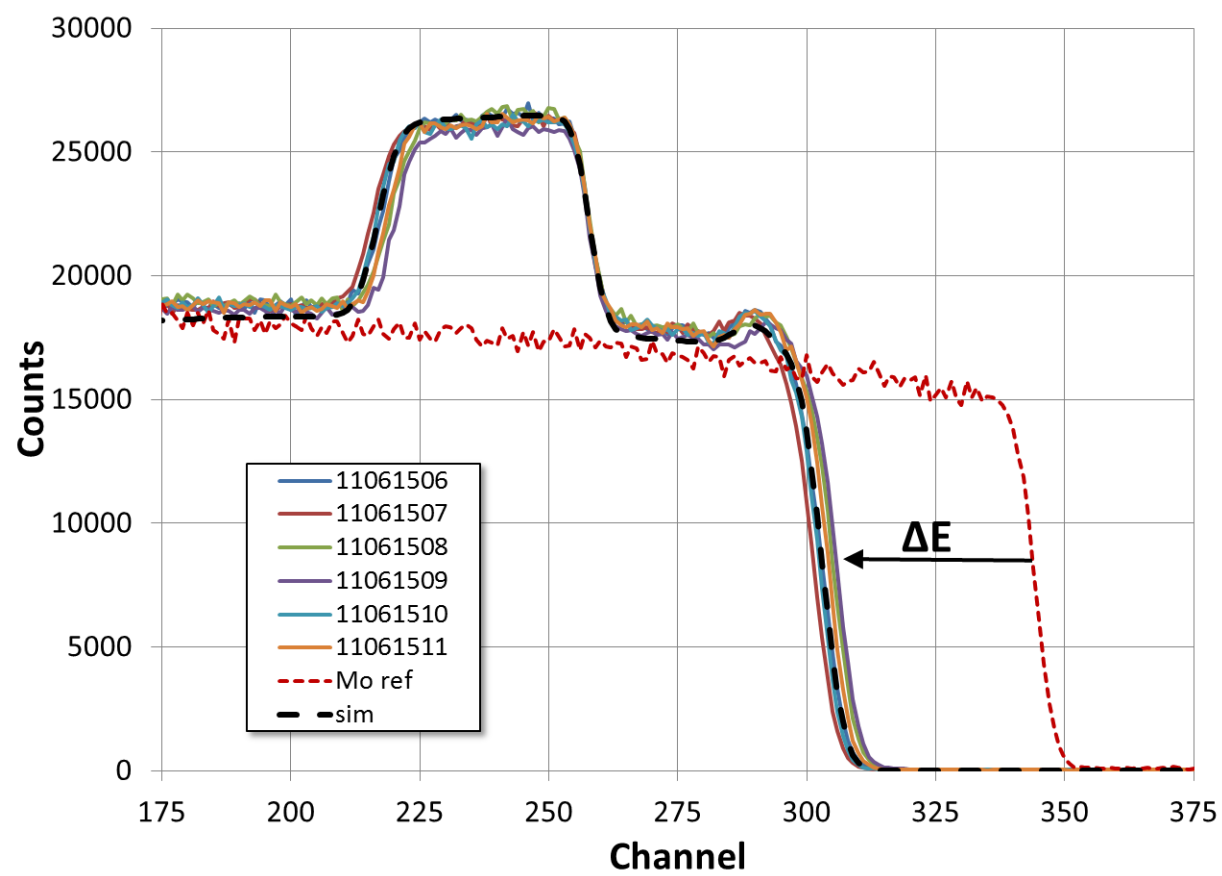


Figure 3. RBS spectra for carbon coated tiles A & L

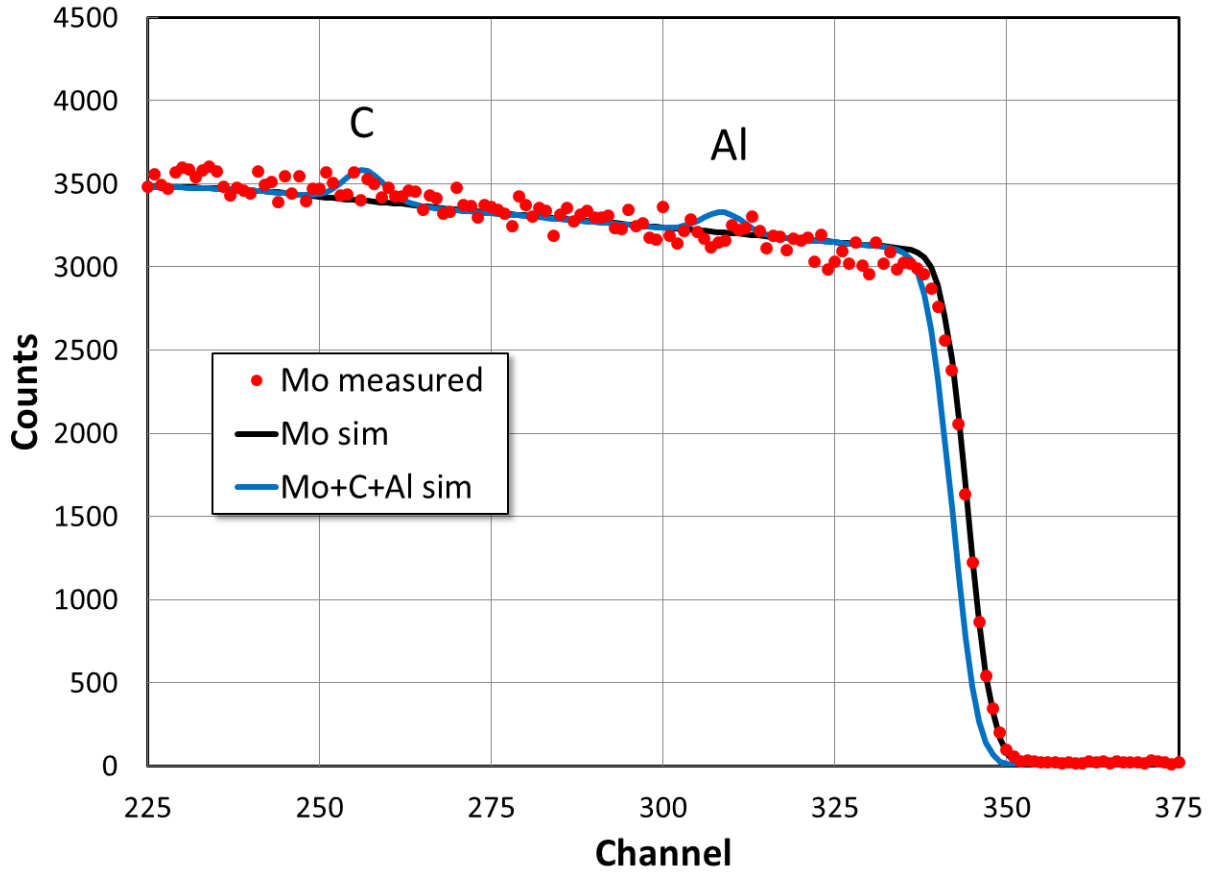


Figure 4. Simulated RBS spectra for 1.5 MeV protons on uncoated Mo (black line) and Mo with  $5 \times 10^{16}/\text{cm}^2$  each of C and Al (blue line), compared to a measured spectrum for bare Mo (red dots). Peaks in the simulation, due to protons scattered from the C and Al, are indicated.

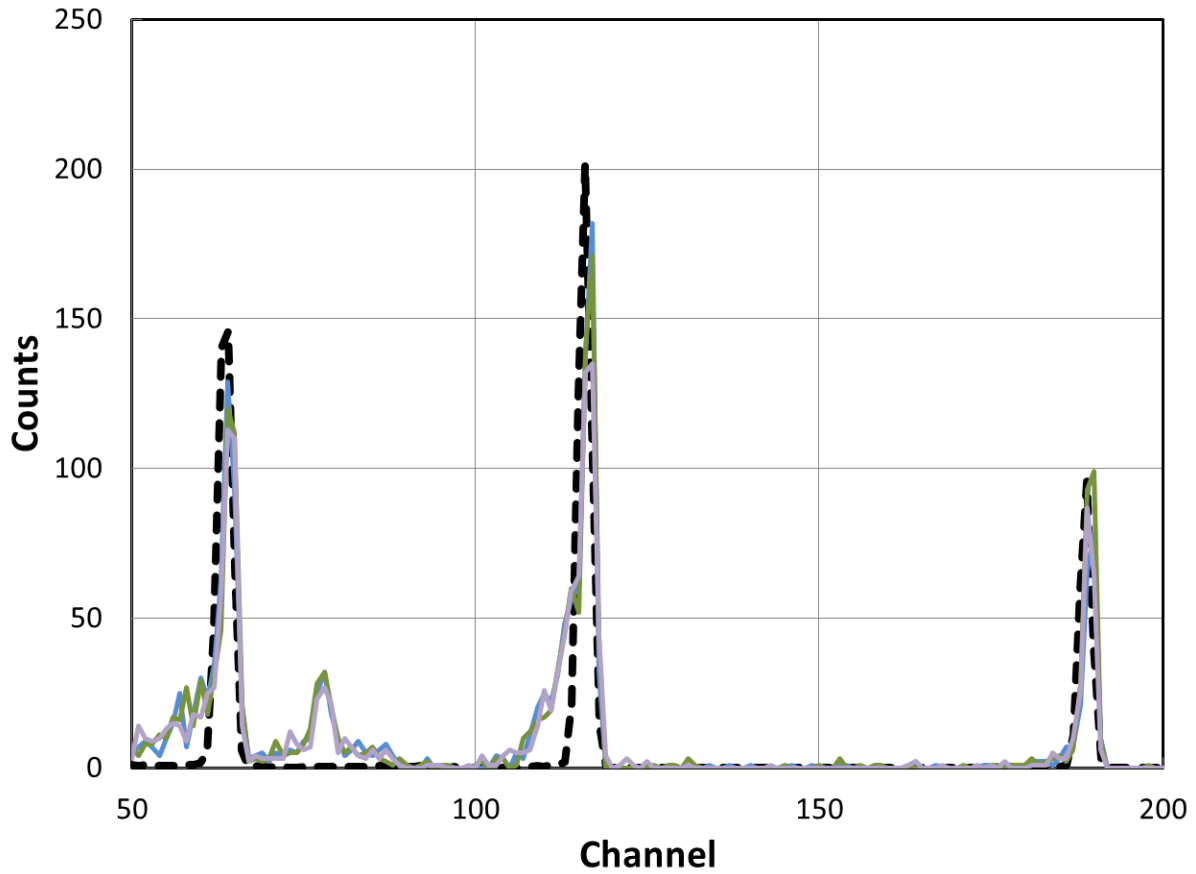


Figure 5. NRA spectra on uncoated Tile C (solid lines) and on a thin-film carbon reference sample (dashed heavy black line). The three large peaks are protons from the  ${}^3\text{He}({}^{12}\text{C},\text{p}){}^{14}\text{N}$  nuclear reaction. The spectrum from the reference, which has  $4.0 \times 10^{18} \text{ C/cm}^2$ , has been scaled down by a factor of 40 for comparison. This measurement shows the tile, in the as-received condition, has  $1.0 \times 10^{17} \text{ atoms/cm}^2$  of carbon which corresponds to about 10 nm.

**Table 1. Summary of film thickness measurements**

<b>Tile</b>	<b>material</b>	<b>Film Thickness (<math>10^{19}</math> atoms/cm<sup>2</sup>)</b>	<b>Film Thickness (micrometer)<sup>†</sup></b>
A	C	$2.298 \pm 0.103$	2.292
B	C	$2.296 \pm 0.166$	2.290
K	C	$2.153 \pm 0.207$	2.148
L	C	$2.239 \pm 0.094$	2.233
E	Al	$1.349 \pm 0.037$	2.238
F	Al	$1.357 \pm 0.009$	2.252
G	Al	$1.346 \pm 0.013$	2.233
H	Al	$1.339 \pm 0.033$	2.222

<sup>†</sup> Mass per unit area divided by density: 2.7gm/cm<sup>3</sup> for Al and 2.0 gm/cm<sup>3</sup> for C.

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